Study On Effect Of Nanosilica Sand Addition On Physical And Mechanical Properties Of Alumina Powder

by

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Final Dissertation submitted in partial fulfillment of the requirements for the Bachelor of Engineering (Hons) (Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the Mechanical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (MECHANICAL ENGINEERING)

Approved by,

(AP Dr. Othman Bin Mamat)

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK June 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources of persons.

(Mohamed Khalil Bin Mohamed Razif)

ABSTRACT

Ceramic Matrix Composites is becoming more and more reliable materials in the industry. Advanced Ceramic Engineering development has been done making the process as well as cost in producing the Ceramic Matrix Composites become lesser and more reliable. The study is being done to observe the effects or differences that occur due to variation of alumina and silica sand nanoparticle to the alumina-silica sand nanoparticles composites. The objective of the study is to establish the effect of silica sand nanoparticles addition on the mechanical and physical properties of alumina based composites. The problem of the study is to find the most optimum or suitable composition in order to enhance the composites usage in the industry. The powder metallurgy technique was used with an uniaxial dry pressing. The green samples were sintered at 1100 °C temperature in argon atmosphere for two, four and six hours of sintering time according to the sample set. It was observed that the green and sintered density of the samples decreased along with the increase in the silica-sand nanoparticles content. The hardness of the sintered composites increased with the increased of silica sand nanoparticles content. The sintered density of the samples was increased with the sintering time up to four hours and decreased at six hours sintering time. The sintered microstructure analysis indicated that the diffusion of silica sand nanoparticles to the pores was formed. The maximum density produced is 3.367 g/cm³. The maximum hardness produced is 70.8 HRB. As a conclusion to this, the experiment proves that different composition of alumina powder and silica sand nanoparticles mixture will produced different mechanical and physical properties of the alumina-silica sand nanoparticles composites produced.

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TABLE OF CONTENTS

CERTIFICATION		i
ABSTRACT		iii
ACKNOWLEDGEME	INT	iv
CHAPTER 1: INTRO	DUCTION	
1.1 Back	ground of Study	1
1.2 Probl	em Statement	2
1.3 Objec	ctive	2
1.4 Scope	e of Study	3
CHAPTER 2: LITERA	ATURE REVIEW	
2.1 Cerar	nic Powder	4
2.2 Silica	(SiO_2)	6
2.3 The S	Silica – Alumina (SiO ₂ – Al ₂ O ₃) System	7
2.4 Powd	er Metallurgy – Uniaxial Dry Pressing	8
2.5 Sinter	ring	9
2.6 Relat	ed Works	11
CHAPTER 3: METHO	DDOLOGY	
3.1 Proje	ct Methodology	15
3.2 Exper	riment Methods	16
3.3 Proje	ct Activities and Planning	17
3.4 Mater	rials	17
3.5 Equip	oment	18
CHAPTER 4: RESUL	T AND DISCUSSION	
4.1 Resul	t and Discussion	20
CHAPTER 5: CONCL	USION AND RECOMMENDATIONS	
5.1 Conc	lusion	32
5.2 Reco	mmendations	33
REFERENCE		34
APPENDIX		36

LIST OF FIGURES

FIGURE	TOPIC	PAGE
2.1	Alumina Powder	5
2.2	Silica sand nanoparticles	7
2.3	Phase diagram of the alumina-silica system	7
3.1	Project Methodology	15
3.2	Mounted Alumina-Silica sand nanoparticles composites	17
3.3	Ball Milling	18
3,4	Autopellet Machine	18
3.5	Sintering Furnace	18
3.6	Density Measuring Instrument	18
3.7	Auto mounting Machine	19
3.8	Grinder and Polisher Machine	19
3.9	FESEM Machine	19
3.10	Hardness Testing Machine	19
4.1	Density versus Percentage of nanosilica in the composites	21
4.2	Microstructure of FESEM analysis for alumina-silica sand	23
	nanoparticles composites with two hours sintering time	
4.3	EDX analysis results for alumina-silica sand nanoparticles	24
	composites samples with two hours sintering time	
4.4	Microstructure of FESEM analysis for alumina-silica sand	25
	nanoparticles composites with four hours sintering time	
4.5	EDX analysis results for alumina-silica sand nanoparticles	26
	composites samples with four hours sintering time	
4.6	Microstructure of FESEM analysis for alumina-silica sand	28
	nanoparticles composites with six hours sintering time	
4.7	EDX analysis results for alumina-silica sand nanoparticles	29
	composites samples with six hours sintering time	
4.8	Hardness versus Percentage of nanosilica in the composites	31

LIST OF TABLES

TABLE	TOPIC	PAGE
2.1	Properties of Alumina, Al ₂ O ₃	5
2.2	Properties of Silica, SiO ₂	6
2.3	Important Binders	8
2.4	Important Lubricants	9
2.5	Sintering Process for Some Ceramic Compositions	10
4.1	Sample's Theoretical, Green and Sintered Densities	20
4.2	Sample's Hardness	30

CHAPTER 1 INTRODUCTION

1.1 Background of Study

Ceramic matrix composites (CMCs) combine reinforcing ceramic phases with a ceramic matrix to create materials with new and superior properties. In ceramic matrix composites, the primary goal of the ceramic reinforcement is to provide toughness to an otherwise brittle ceramic matrix [12] or in other words to overcome the intrinsic brittleness and lack of reliability of monolithic ceramics, with a view to introduce ceramics in structural parts used in severe environments such as rocket and jet engines, gas turbines for power plants, heat shields for space vehicles, fusion reactor first wall, aircraft brakes, and heat treatment furnaces, etc [13].

The desirable characteristics of CMCs include high-temperature stability, high thermal shock resistance, high hardness, high corrosion resistance, light weight, nonmagnetic and nonconductive properties, and versatility in providing unique engineering solutions. The combination of these characteristics makes ceramic matrix composites attractive alternatives to traditional processing industrial materials such as high alloy steels and refractory metals [12]. It is generally admitted that the use of CMCs in advanced engines will allow an increase of the temperature at which the engine can be operated and eventually the elimination of the cooling fluids, both resulting in an increase of yield [13].

Although CMCs are promising thermostructural materials, their applications are still limited by the lack of suitable reinforcements, processing difficulties, sound material data bases, lifetime and cost [13].

1.2 Problem Statement

1.2.1 Problem Identification

The alumina powder and silica sand nanoparticles mixture may produce Ceramic Matrix Composite which is alumina-silica sand nanoparticles composite. The properties of this alumina-silica sand nanoparticles composite depend on the quantity or the percent composition of each alumina and silica sand nanoparticles materials. The main problem here is to determine the mechanical and physical properties of alumina-silica sand nanoparticles composite base on the different in the composition of alumina and silica sand nanoparticles materials.

1.2.2 Significance of the Project

Ceramic Matrix Composite is developing from time to time. The Ceramic Matrix Composite has been reinforced in order to achieve the primary goal of ceramic reinforcement which is to enhance the properties of the original materials such as its toughness, density and hardness [12]. This project will not only establish new data for the produced materials. The most important part of this project is to expose the methods, experiments and manufacturing processes to the UTP students in order for them to understand further the theoretical knowledge from the courses they taken.

1.3 Objective

The objective of the study is to establish the effect of silica sand nanoparticles addition on the mechanical and physical properties of alumina based composites.

1.4 Scope of Study

In this writing, the writer will study on the effect of silica sand nanoparticles addition on the alumina especially on the product mechanical and physical properties. The study will be varied by differentiating the composition of the alumina and silica sand nanoparticles in creating samples of alumina-silica sand nanoparticles composites through powder metallurgy method. The samples will have different mechanical and physical properties. For example, sample 1 will be composed of 90% alumina and 10% silica sand nanoparticles.

The mechanical properties that will be observed are the hardness of the material. The physical properties that will be observed are the green density and the sintered density as well as the microstructure of the material.

CHAPTER 2 LITERATURE REVIEW

2.1 Ceramic Powders

The boundaries between powder metallurgy and ceramics are no longer strictly defined; some information on ceramic powders is useful in the context of powder metallurgy. The synthesis of ceramic powders employs a wide variety of methods, yielding powders of very different particle size distribution and specific surface. The products also may have different purity, compatibility, sinterability, chemicals and phase composition [1].

However the methods of producing ceramic powders either using the conventional methods or new methods, both of them implement an improvement towards the purity of the ceramic powders besides controlling the grain size and the distribution of the ceramic powders. The improvements will also slowly bringing the technologies of producing ceramic powders to the production of the ceramic powders nanoparticle which are said to be advantageous upon the ceramics consolidation [4].

2.1.1 Alumina Powder (Al₂O₃)

Alumina (Al₂O₃) is by far the most important oxide ceramic and its powder is produced on a very large scale and to a wide variety of specifications. Almost 35 million tonnes per year are produced throughout the world, of which 93 % is used as 'smeltering grade material' for the production of aluminum. The remainder is mostly calcined and milled for use in abrasive and polishing applications and in the production of refractories and ceramics. All qualities are produced from Bauxite via the Bayer process which is a wet alkaline route, separating the Al_2O_3 from Fe_2O_3 and other oxides by forming sodium aluminate except for powders with highest purity (99.99 %). This is transformed by hydrolysis into Al (OH) ₃ and subsequently calcined to Al_2O_3 . The highest purity aluminas are predominantly obtained by the decomposition of high purity aluminumbased salts [1].

The properties of Al₂O₃ are as per stated in the table below:

NO	PROPERTIES	DETAILS
1	Molecular Formula	Al_2O_3
2	Molar Mass	101.96 g/mol
3	Appearance	White solid, very hygroscopic.
4	Odor	Odourless.
5	Density	3.59 - 4.1 g/cm ⁻³
6	Melting Point	2072 °C
7	Boiling Point	2977 °C
8	Solubility in Water	Insoluble.

Table 2.1: Properties of Alumina, Al₂O₃ [14].

Besides the properties above, Al_2O_3 also have high hardness which is 2100 knoop hardness, good electrical insulator, have high thermal conductivity, high specific heat which is 775 J / kg-K and have tensile strength in the range of 337 to 551 MPa [2].



Figure 2.1: Alumina Powder

2.2 Silica (SiO₂)

SILICA is the most abundant mineral found in the crust of the earth. It forms an important constituent of practically all rock-forming minerals. It is found in a variety of forms, as quartz crystals, massive forming hills, quartz sand (silica sand), sandstone, quartzite, tripoli, diatomite, flint, opal, chalcedonic forms like agate, onyx etc., and in with numerous other forms depending upon colour such as purple quartz (amethyst), smoky quartz, yellow quartz or false topaz (citrine), rose quartz and milky quartz. Only pure quartz crystal or rock crystal, untwined, clear, free from any inclusion, has an important property. Silica sand will be used to establish the mechanical and physical properties of alumina-nano silica composite of this project [16]. The Tronoh silica sand will be processed to nano-silica sand (60-80nm) using the dry ball milling.

The properties of SiO_2 are as per stated in the table below:

NO	PROPERTIES	DETAILS
1	Molecular Formula	SiO ₂
2	Molar Mass	60.0843 g/mol
3	Appearance	White Powder.
4	Density	2.634 g/cm^3
5	Melting Point	1650 (± 75) °C
6	Boiling Point	2230 °C
7	Solubility in Water	0.012 g/100 mL

Table 2.2: Properties of Silica, SiO₂ [16].

 SiO_2 also have other properties such as hardness of 800 knoop hardness, low thermal conductivity, high specific heat which is 740 J / kg-K and have tensile strength of 104 MPa [2].



Figure 2.2: Silica sand nanoparticles

2.3 The Silica – Alumina (SiO₂ – Al₂O₃) System

Commercially, the silica-alumina system is an important system since the principal constituents of many ceramic refractory are these two materials. Figure shows the $SiO_2 - Al_2O_3$ phase diagram. The polymorphic form of silica is stable at these temperature is termed *cristobalite*. Silica and alumina are not mutually soluble in one another and proven by the absence of terminal solid solutions at both extremities of the phase diagram [2].



Figure 2.3: Phase diagram of the alumina-silica system [17]

2.4 Powder Metallurgy – Uniaxial Dry Pressing

Uniaxial dry pressing is a basic process in powder metallurgy which is used in producing ceramics samples with simple shape. The process is cheap and simple compare to other process and has been successfully automated. The process only required powders of the material to be pressed to be inserted into the die. The powder is preferred in the form of granulated. The powder is then pressed at one or both entrance of the die to press the powder into the wanted shape. The stages in the process are, sliding and rearrangement of particles, particle fragmentation and the elimination of the pores. However, the particle fragmentation stage is often left out in the pressing of ceramics powder without binders. Binders are used to impart plasticity to powders while the lubricants are to be used to improved particles sliding [4].

Table 2.3 listed down a few important binders that are suitable and can be used for ceramics materials.

Trade name	Composition	Materials	Amount (wt %)	Process	Pressure Range (MPa)	Burnout Range (°C)
PVA	Poly(vinyl alcohol)	Al ₂ O ₃ , ZrO ₂ , MgAl ₂ O ₄	0.1-2	Slip casting, extrusion, dry pressing, tape casting.	7-70	250-400
PVB (B-76)	Poly(vinyl butyral)	Al ₂ O ₃ , ZrO ₂	2-15	Tape casting, dry pressing, extrusion.	70-400	250-300
Cimarec	Tertiary amide polymer	Oxides		Dry pressing	2	<u></u>
Pyrofine PV	Polysilazane	Advanced ceramics	-	Dry pressing	2	1793 1
Methocel	Methicellulose	Al ₂ O ₃ , ZrO ₂	1.5-3	Extrusion , dry pressing	35	250-450
Polyox	Resin	Al ₂ O ₃	1.5	Dry pressing	35	200-300
Carbowax 20M	Poly(ethylene glycol)	Oxides	1-3	Dry pressing	20-70	250-350
Darvan	Sodium	Oxides	<u> </u>	Spray drying		
No.7	polyelectrolyte Cellulose Ethers	Oxides	7-20	Extrusion		250-350
SR350/SR 355	Silicone	Oxides	-	Injection molding	Ξ.	900
Ross wax	Paraffin wax	Oxides	3	Dry pressing, injection molding	20-35	200-400

Table 2.3: Important Binders [4].

Table 2.4 listed down a few important lubricants that are suitable and can be used for ceramics materials.

Function	Trade Name	Compositions	Materials	Amount (wt%)	Process	Burnout Range (°C)	Company
Lubricants	<u>a</u> :	Oleic acid	All	0.5-1	Dry pressing	350-375	1070
	Neofat	Tall oil	All	0.5-1	Dry pressing	350-375	-
	Dry lubricants	Graphite.BN. talc (plate form)	Al ₂ O ₃ , ZrO ₂ , SiC	1	High temp. Process	550 and up (BN and talc remain)	(-)
	-	Butyl Stearate	All	0.1-0.5	Dry pressing	275-500	121
	2	Polyglycols	All	1	2	2	3 <u>8</u> 6
Flocculants	Glucoplus 110	Pre gelled, cationic corn starch	For colloidal silica and alumina binder	-	-	-	Wesbond
Defoaming agents	-	Tributyl phosphate	All	-	-	250-500	12
26.081	DB-31	Silicone-based	All		-	250-500	
	DB-33	Silicone-based	All	9 1 1	-	250-500	100
	Foamaster	-	All		=	-	Nopeo

Table 2.4: Important Lubricants [4].

2.5 Sintering

Sintering is the heat treatment process in which the powder or porous materials that has been formed, is converted to a useful solid. The sintering processes commonly are divided into several categories which depending on the type of the system to be sintered. One of the categories is for a pure, single-phase, polycrystalline material. The sintering in this category can be achieved by heating the consolidated mass of particles which referred to as green body or powder compact, to the temperature 50% -80% of its melting temperature. For Al_2O_3 which has the melting temperature of 2073 °C, the sintering temperature generally will be between 1400 °C and 1650 °C. The powder will not melt, instead, the joining of the particles and reduction of porosity will occur through atomic diffusion in the solid state. This type of sintering process is known as solid-state sintering [3].

For many polycrystalline ceramics, they undergone the liquid–phase sintering method due to the characteristic that wanted to be obtained was difficult to obtain using the solid-state sintering method. The liquid phase provides a high-diffusivity path for transport of matter into the pores to produce densification. However it is insufficient to fill the porosity by itself. Another solution for the polycrystalline ceramics was by using the pressure sintering where external pressure is exerted to the body during the heating in solid-state or liquid-phase sintering. Other sintering processes for some ceramic composition can be found in the table below [3]:

NO	COMPOSITION	SINTERING PROCESS
1	Al ₂ O ₃	Solid-state sintering with MgO additive.
		Liquid-phase sintering with silicate glass.
2	MgO	Liquid-phase sintering with a silicate glass.
3	Si ₃ N ₄	Liquid-phase sintering with oxide additive under nitrogen
		gas pressure or under an externally applied pressure.
4	SiC	Solid-state sintering with B and C additives.
		Liquid-phase sintering with Al, B and C or oxide additives.
5	ZnO	Liquid-phase sintering with Bi ₂ O ₃ and other oxide
		additives.
6	BaTiO ₃	Liquid-phase sintering with TiO ₂ -rich liquid.
7	Pb (Zr,Ti)O ₃	Sintering with a lead-rich liquid phase; hot pressing.
	(PZT)(Pb,La)(Zr,Ti)	
	O ₃ (PLZT)	
8	ZrO ₂ /(3-10 mol %	Solid state sintering.
	Y ₂ O ₃)	
9	Mn-Zn and Ni-Zn	Solid-state sintering under a controlled oxygen atmosphere.
	ferrites	
10	Porcelain	Vitrification.
11	SiO ₂ gel	Viscous sintering.

Table 2.5: Sintering Process for Some Ceramic Compositions [3].

2.5.1 Solid-State Sintering

The driving force of solid-state sintering depends on the tendency of the particles in reducing their surface energies. However there are alternatives of this which are the grain growth or coarsening without pore removal and the densification with pore shrinkage. Both of the phenomena are said to be competitive and interactive [4].

2.6 Related works

a) M. Mujahid, M.I. Qureshi, M. Islam and A.A. Khan, "Processing and Microstructure of Alumina-Based Composites"

In this paper, the author focused on the powder structure and its effect on the sintering tendency of alumina-based ceramic systems, which is Al_2O_3 -SiO₂ and Al_2O_3 -ZrO₂ in improving its mechanical strength and fracture toughness. The author experiment was carried out firstly by particle characterization of each constituent using Scanning Electron Microscope (SEM). The experiment continued with the preparation of composite mixture by weighing the constituent powders and mixed thoroughly in producing aqueous suspension. Sodium phosphate was added as a dispersant. The mixture was simultaneously heated using a magnetic hot plate until it dried. The dried mass was mixed with a plasticizer and ground one more time. The mixture was then cold press and followed by sintering at a maximum temperature of 1500 °C. The finding of the author was the best densification was obtained for mixture that using very fine and deagglomerated alumina powders which the density of the samples contain 10 % silica sand addition to alumina powder is 3.47 g/cm³[6].

b) **B.** Sudhir, Atul H. Chokshi, "Creep characterization of alumina particulate reinforced alumino-silicate glass"

In this paper, the author doing the study on compressive creep of alumino-silicate glass reinforced with alumina powders of 0.2μ m particle size as a function of volume fraction. It is showed that there was a significant increase in the creep resistance by about an order of magnitude in 20 vol. % composite. The author also characterized the effect composites reinforced with 20 vol. % of 1.2 and 6.5 μ m. The author found that the composite with 1.2 μ m particle size reinforcement gave the highest creep resistance. All the composites exhibited Newtonian viscous creep and showed no strain hardening even at strain up to 10 % [7].

c) Kangning Sun, Aimin Li, Xuemin Chi, Yangsheng Yin, Peiwu Li, "Sintering technology research of Fe₃Al/Al₂O₃ ceramic composites"

In this paper, the author focused on the effects of using different sintering technologies on the microstructure of the Fe₃Al/Al₂O₃ ceramic composites. The author experiment consisted of 6 samples which each sample is differentiate from each other in term of its composition, the hot press pressure which is 20-30MPa or the sintering temperature which is 1300-1450 °C. The fabrication process started with the preparation of Fe₃Al powder using the mechanical alloying method. Then Fe₃Al and Al₂O₃ powder are mixed by ball milling. The composite powder is put into a graphite mould and sintered by hot pressure. The author finding on the result of the experiment indicate that Fe₃Al/Al₂O₃ composites with fine grains, compact structure and good mechanical properties can be obtain at 1400 °C of sintering temperature [8].

d) Wilson Acchar^I, José Roberto Bezerra da Silva, "Surface characterization of alumina reinforced with niobium carbide obtained by polymer precursor"

In this paper, the author study the characterization of alumina reinforced with niobium carbide. The author experiment is crucial for the use of cutting tools surface hardness and wear resistance. The samples which contained fixed concentration of 60 wt (%) of polysiloxane and mixture of alumina powder and metallic niobium were homogenized and unaxially warm pressed as 80 °C. The samples are subsequently pyrolized in flowing argon atmosphere at 1200, 1400 and 1500 °C. The analyses are carried out using X-ray, infrared and scanning electron microscope. The results indicate that the phases on the surface are depending to the niobium/carbon ration of the materials [9].

e) Wilson Acchar^I, Carlos Alberto Cairo^{II}, Ana Maria Segadães^{III}, "*TEM study of a hot-pressed Al*₂O₃-*Nbc composite material*"

In this paper, the author studies the effect in reinforcing Al2O3 with refractory carbides to the mechanical properties such as hardness, fracture toughness and wear resistance. Al₂O₃-NbC composite was produced and were unaxially hot-pressed at 1650 °C in inert atmosphere. Their mechanical properties were analyzed. The results obtained shows that the mechanical properties of Al₂O₃-NbC composite produced are comparable to other carbide system and from the microstructure analysis, the author obtained the location niobium carbide particle mainly positioned which is in the grain boundaries of alumina grains which gives the evidence of the "pinning effect" by NbC particles [10].

f) Marie C. Willson, H. J. Edrees* and Kerr, "Fabrication of Composite Ceramics Using Dry mixing Process"

In this paper, the author studies the potential route for the production of randomly short fibre CMCs. The composite system studied was a silicon nitride matrix reinforced with silicon carbide whiskers. The route involved was dry mixing, production of soft agglomerates, uniaxial and pressing and densification by sintering in overpressure. Comparative strength are done using biaxial testing method and the results obtained was this route can be considered to be a potential route for the production but required further improvement [11].

CHAPTER 3 METHODOLOGY

3.1 Project Methodology

Figure 3.1 shows the overall project flow.



Figure 3.1: Project Methodology.

Figure 3.1 consisted of all the activities that will be done within the two parts of the project. The problem statement and literature review process will be done during FYP 1 period. The problem statement will consist of the important item that required to be understood prior to conduct the research. The literature review is part of the research that will be conducted after understanding the problem statement. Literature review usually will be based on the journal of the experiment and research work that had been done by other researcher.

The second parts of the project consist of the experiment, testing, result & analysis, discussion, conclusion and finally recommendation. However, minor literature review will still be commenced in order to make sure the journal being used in the report are up to date. The project activities and planning will be discussed in the next part of this chapter.

3.2 Experiment Method

Alumina powder with 63 µm particle size and silica sand nanoparticle originated from Tronoh, Perak, Malaysia are being used in this project. Both of the constituent have been prepared by manufacturers. The constituent was mix together by using ball milling for 1 hour. The mixture was then added with 1% of paraffin wax which depends on the weight of the mixture as the binding agent. The mixture was than being compact using the autopallet machine and 13 mm metallic mould at 145 MPa force. The composition of the sample to created are Pure Al₂O₃, 5% silica sand nanoparticle, 10% silica sand nanoparticle, 15% silica sand nanoparticle and 20% silica sand nanoparticle Al₂O₃ based composites. The process follows with the green density measurement of the samples using the Archimedes method. Then the sample was split into three set and sintered at 1100 °C at sintering time of 2 hours, 4 hours and 6 hours respectively in Argon atmosphere. The heating and cooling rate during the sintering process was 10 °C/min. After the sintering process, the sintered density was measured using the Archimedes method. The samples were then mounted by PhenoCure for grinding and polishing purposes. The polished samples were then being analyzed using Field Emission

Scanning Electron Microscope (FESEM) to see its microstructure. Finally, the sample will undergo the hardness testing using hardness testing machine with Rockwell scale.



Figure 3.2: Mounted Alumina-Silica sand nanoparticles composites

3.3 Project Activities and Planning

The project activities that has and going to be done are as per stated in the Gantt Charts which are being attached as appendixes at the end of this report. The Appendix 1 and 2 showed the Gantt chart for FYP1 and FYP 2.

3.4 Materials

The material that will be used is alumina powder and silica sand nanoparticles. This alumina powder will be reinforced with Tronoh silica sand nanoparticles which will be produced by using ball milling. The size of Alumina powder particle used are 63 μ m while the Tronoh silica sand nanoparticles was estimated to be within the range of 60-80 nm.

3.5 Equipment

The equipment used in this study included: Ball milling, Autopellet Press Machine, Sintering furnace, density measurement instrument using Archimedes Method, auto mounting machine, grinder and polisher machine, Field Emission Scanning Electron Microscope (FESEM) and hardness testing machine. Some of the machines can be found in the following page.





CHAPTER 4 RESULT AND DISCUSSION

4.1 Result and Discussion

4.1.1 Density

Table 4.1 above shows the theoretical, green and three sintered density for the samples with the composition stated.

Samples Composition			Density (g/cm	3)	
Samples Composition	Theoretical	Green	Sintered 2hr	Sintered 4hr	Sintered 6hr
Pure Alumina	3.980	2.624	3.274	3.367	3.168
Alumina + 5% Silica sand nanoparticle	3.881	2.570	3.195	3.236	3.053
Alumina + 10% Silica sand nanoparticle	3.787	2.456	3.108	3.047	2.814
Alumina + 15% Silica sand nanoparticle	3.697	2.245	2.747	3.167	2.644
Alumina + 20% Silica sand nanoparticle	3.611	1.984	2.699	2.312	2.483

Table 4.1: Sample's Theoretical, Green and Sintered Densities

All the density excluding the theoretical density was measured using the Archimedes Method. As for the theoretical density, the results are calculated based on the rules of mixture. The formula used in calculating the theoretical density is as follow:

$$\rho = 100 / ((W_f / \rho_f) + (W_m / \rho_m))$$
 [2].

Where ρ is the Theoretical Density, W f and W m is the weight percentage for the particulate (SiO₂) and matrix (Al₂O₃) respectively while ρ f and ρ m is the density of the particulate (SiO₂) and the matrix (Al₂O₃) respectively. ρ f are taken to be 2.634 g/cm³ [2] and ρ m are taken to be 3.980 g/cm³[2].

Figure 4.1 shows the graph generated from the results gained in table 4.1. The theoretical values calculated have the highest density compared to other experimental values and are marked with dark blue line. The trend of the theoretical density is decreasing as the percentage of silica sand nanoparticles increased. The trend of the theoretical density decreased is due to the effect of decreasing amount or composition of alumina powder in the composites. Based on the literature review done on the alumina powders and silica sand pure density, it shows that the density of pure alumina is higher which is in the range of 3.59 to 4.1 g/cm^3 [14] while the density of the silica sand are taken as 2.634 g/cm^3 [2,16]. So, when the composition of silica sand increased, the density will decreased in accordance to the theoretical density formula stated previously.



Figure 4.1: Density versus Percentage of nanosilica in the composites

Figure 4.1 shows the green density which represented by red line is situated at the bottom of other lines. This is due to the porosity that occurred inside the samples due to some strong particles of alumina and silica–sand nanoparticles that cannot be brake into smaller size particles as the samples were produced only by dry pressing process. However, the trends of the samples are the same as the trend calculated theoretically.

The other three lines represent the sintered density of three set of samples. The samples are varied from each other based on the sintering time it takes during the sintering process. The green lines represent the sintered density for 2 hours samples, while the violet and pale blue line is representing the sintered density for 4 hours and 6 hours sample respectively. As per in figure 4.1, the three sintered density lines generally follows the theoretical density line trend. However, at the composition of alumina powder reinforced with 15% of silica sand nanoparticles, there are a little decrease and increase of the sintered density value for 2 hours sample and 4 hours sample respectively. These effects are might be due to the porosity size that occurred inside of the samples.

The three sintered density line however, if being observed in the term of the sintering time taken for each samples, it shows that the longer the sintering times, the denser the samples become. However density only increased up to four hours sintering time. The density of the samples decreased at the sintering time of six hours. The phenomena might be due to the flow of trapped gas within the pores site exiting the samples through small opening when the gas particle kinetic energy is sufficient and due to the lost in water composition in the samples of six hours sintering.

4.1.2 Microstructure Analysis

The microstructure of the alumina-silica sand nanoparticles composites sample with 5%, 10%, 15% and 20% silica sand nanoparticles additions are analyzed and obtained using the FESEM analysis at 1000X resolution. The results of the analysis are as follows.

4.1.2.1 FESEM analysis for alumina-silica sand nanoparticles composites samples with 5%, 10%, 15% and 20% silica sand nanoparticles additions sintered at 1100 °C with two (2) hours sintering time.

Figure 4.2 shows the microstructure of the samples that been sintered at 1100 °C for two hours sintering time.



Figure 4.2: Microstructure of FESEM analysis for alumina-silica sand nanoparticles composites with (a) 5%, (b) 10%, (c) 15% and (d) 20% silica-sand nanoparticles additions at 1100 °C with two (2) hours sintering time

Figure 4.2 shows that there are good interfacial integrity between the alumina powder and the silica sand nanoparticles. It can be observed that throughout the samples that there is homogeneous distribution of the silica-sand nanoparticles due to the filling of the pores by these silica-sand nanoparticles regardless the size of the pores. The EDX analysis of the alumina-silica sand nanoparticles composites sample with 5%, 10%, 15% and 20% silica sand nanoparticles additions for sintered sample at 2 hours sintering temperature are conducted. The results of the analysis are shown as in Figure 4.3.





Figure 4.3: EDX analysis results for alumina-silica sand nanoparticles composites samples with (a) 5%, (b) 10%, (c) 15% and (d) 20% silica-sand nanoparticles additions at 1100 $^{\circ}$ C with two (2) hours sintering time

Figure 4.3 shows the microstructure of the alumina-silica sand nanoparticles composites samples and the composition that the samples contained. As observed from figure 4.3a to figure 4.3d, the quantity of silica sand nanoparticles are to be found increased.

4.1.2.2 FESEM analysis for alumina-silica sand nanoparticles composites samples with 5%, 10%, 15% and 20% silica sand nanoparticles additions sintered at 1100 °C with four (4) hours sintering time.

Figure 4.4 shows the microstructure of the samples that been sintered at 1100 °C for four hours sintering time.





Figure 4.4: Microstructure of FESEM analysis for alumina-silica sand nanoparticles composites with (a) 5%, (b) 10%, (c) 15% and (d) 20% sand nanoparticles additions at $1100 \,^{\circ}$ C with four (4) hours sintering time

Figure 4.4 shows that there are good interfacial integrity between the alumina powder and the silica sand nanoparticles as per in samples for two hours sintering time. However, it can be observed that throughout the samples that the homogeneous distribution of the silica-sand nanoparticles due to the filling of the pores by these silicasand nanoparticles is much better compared to the two hours samples. The reinforcement in this samples filled more pores area.

The EDX analysis of the alumina-silica sand nanoparticles composites sample with 5%, 10%, 15% and 20% silica sand nanoparticles additions for sintered sample at 4 hours sintering temperature are conducted. The results of the analysis are shown as in Figure 4.5.





Figure 4.5: EDX analysis results for alumina-silica sand nanoparticles composites samples with (a) 5%, (b) 10%, (c) 15% and (d) 20% silica-sand nanoparticles additions at 1100 °C with four (4) hours sintering time.

Figure 4.5 shows the microstructure of the alumina-silica sand nanoparticles composites samples and the composition that the samples contained. As observed from figure 4.5a to figure 4.5d, the quantity of silica sand nanoparticles are to be found increased.

4.1.2.3 FESEM analysis for alumina-silica sand nanoparticles composites samples with 5%, 10%, 15% and 20% silica sand nanoparticles additions sintered at 1100 °C with six (6) hours sintering time.

Figure 4.6 shows the microstructure of the samples that been sintered at 1100 °C for six hours sintering time.



Figure 4.6: Microstructure of FESEM analysis for alumina-silica sand nanoparticles composites with (a) 5%, (b) 10%, (c) 15% and (d) 20% sand nanoparticles additions at 1100 $^{\circ}$ C with six (6) hours sintering time

Figure 4.6 shows that there are better interfacial integrity between the alumina powder and the silica sand nanoparticles compared to the samples for two hours and four hours sintering time. The homogeneous distribution of the silica-sand nanoparticles due to the filling of the pores by these silica-sand nanoparticles is the best compared to the two hours and four samples. The reinforcement in this sample filled much more pores area. The EDX analysis of the alumina-silica sand nanoparticles composites sample with 5%, 10%, 15% and 20% silica sand nanoparticles additions for sintered sample at 6 hours sintering temperature are conducted. The results of the analysis are shown as in Figure 4.7.





Figure 4.7: EDX analysis results for alumina-silica sand nanoparticles composites samples with (a) 5%, (b) 10%, (c) 15% and (d) 20% sand nanoparticles additions at 1100 $^{\circ}$ C with Six (6) hours sintering time

Figure 4.7 shows the microstructure of the alumina-silica sand nanoparticles composites samples and the composition that the samples contained. As observed from figure 4.7a to figure 4.7d, the quantity of silica sand nanoparticles are to be found increased.

4.1.3 Hardness

Table 4.2 shows the data of hardness for the samples sintered at two, four and six hours of sintering times with the composition stated. The data are measured using Hardness Rockwell unit (HRB).

Samples Composition	H	Hardness (HRB)
Samples Composition	Sintered 2hr	Sintered 4hr	Sintered 6hr
Pure Alumina	41.8	43.8	45.9
Alumina + 5% Silica sand nanoparticle	44.8	48.6	51.1
Alumina + 10% Silica sand nanoparticle	53.7	57.5	58.3
Alumina + 15% Silica sand nanoparticle	60.8	62.7	66.0
Alumina + 20% Silica sand nanoparticle	62.6	66.1	70.8

Table 4.2: Sample's Hardness

Figure 4.8 is the graph representing the hardness versus percent silica-sand nanoparticles percentage. As per shown in the graph, the hardness of alumina-silica sand nanoparticles composites changes with change in the percentage of silica-sand nanoparticles addition. The hardness of the alumina-silica sand nanoparticles composites increased with the increase of the percentage of silica-sand nanoparticles. This shows that the silica-sand nanoparticles which act as reinforcement enhance the hardness of the alumina-silica sand nanoparticles. This might be the effect of the filling of pores by the silica-sand nanoparticles.



Figure 4.8: Hardness versus Percentage of nanosilica in the composites

Figure 4.8 also shows that the hardness reacts with the sintering temperature. As per shown in the graph, the hardness of the alumina-silica sand nanoparticles composites increase as the sintering time increase. This indicates that there might be good interfacial integrity as well as diffusion between the particles within the alumina-silica sand nanoparticles composites.

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The results obtained from the experiments shows that there are decreasing trend of the density when the composition of silica sand nanoparticles was added into the alumina powder. However, in term of the sintering density, the trend increased when the sintering time increased up to four hours and decreased at six hours sintering time. The maximum density produced is 3.367 g/cm³. Based on the FESEM and EDX analysis, it can be observed that the silica sand nanoparticles are diffuse in the porous sites of composites. This created an improvement in the mechanical and physical properties of the alumina-silica sand nanoparticles composites produced. The hardness of the alumina-silica sand nanoparticles composites produced is also affected. The hardness of the alumina-silica sand nanoparticles composites produced, increased with the increased in silica sand nanoparticles addition to alumina powder. The hardness also increased along with the increased of the sintering time. The maximum hardness produced is 70.8 HRB. As a conclusion to this, the experiment proves that different composition of alumina powder and silica sand nanoparticles mixture will produced different mechanical and physical properties of the alumina-silica sand nanoparticles composites produced.

5.2 Recommendations

5.2.1 First Recommendation

The first recommendation that can be done is by producing a mold in which a powder metallurgy dog bone sample of the alumina-silica sand nanoparticles can be produced. This dog bone shape samples are to be used in order to test or analyze the alumina-silica sand nanoparticles composites tensile strenght. The mold sample is as in appendix 3 and 4.

5.2.2 Second Recomendation

The second recommendations that can help in the further studies of the materials are by using higher temperature during sintering process. As stated in the sintering section under literature review chapter of this report, the suitable range of the sintering temperature are between 1400 °C until 1650 °C while the one being used in the experiment are 1100 °C due to limitation of the furnace being used. Although the experiment can be accepted as successful, by increasing the sintering temperature into the range mentioned above, the result of the experiment may be improved.

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APPENDIX

APPENDIX 1 : FYP 1 GANTT CHART

TITLE: STUDY ON EFFECT OF NANOSILICA SAND ADDITION ON PHYSICAL AND MECHANICAL PROPERTIES OF ALUMINA POWDER

ACTIVITIES	1	2	3	4	5	6	7	8	9		10	11	12	13	14
Meetings with SV															
Prelim Research Work															
Research on Powder Metallurgy															
Research on related journal															
Reseach on Alumino-Silicate System										\sim					
										al					
Research Work Phase 1										Le Le					
Reseach on Background of Study										B					
Research on Tronoh Nanosilica										<u> </u>					
Research on Alumina Properties										e					
Research on Nanosilica addition effects										S					
Research on Suitable Methodology										Je					
										Ц.					
Report Writing										Se					
Prelim report					Δ										
Progress report								Δ		10					
Interim report										\geq				Δ	
Seminar/Briefing															
Oral Presentation															
Preparation															Δ



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TITLE: STUDY ON EFFECT OF NANOSILICA SAND ADDITION ON PHYSICAL AND MECHANICAL PROPERTIES OF ALUMINA POWDER

ACTIVITIES 2 3 4 5 6 7 8 9 10 11 12 13 14 1 Meetings with SV Project Work Phase 1 Comfirming Methodology Conducting Experiment Conducting Physical Testing Result Analysis and Discussion Brake Project Work Phase 2 Conducting Experiment Conducting Mechanical Testing Semester Result Analysis Conclusion and Recommendation Report Writing Progress report 1 Δ Progress report 2 Δ Disertation Final Report Mid Disertation Final Report (Hardbound) Δ Poster Exhibition Δ Seminar/Briefing Δ Oral Presentation Preparation Δ

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APPENDIX 3: SINGLE MOLD DESIGN FOR TENSILE STRENGH ANALYSIS



APPENDIX 4: DOUBLE MOLD DESIGN FOR TENSILE STRENGH ANALYSIS

